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Description

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This invention relates generally to polyols produced using double metal cyanide complex catalysts and, more specifically, to a process for improving the reactivity of double metal cyanide complex catalyzed polyols.

The use of double metal cyanide complex ("DMC") catalysts in the preparation of polyols having low ethylenic unsaturation is well-established in the art, as illustrated, for example, by US-A-3,829,505. The polyole prepared using these catalysts can be fabricated to have a higher molecular weight and/or a lower amount of end group unsaturation than can be prepared using commonly-employed potassium hydroxide ("KOH") catalysts. The "505 patent discloses that these high molecular weight polyol products are suitably reacted with polyieocyanates to prepare rigid or flexible polyurethanes.

Unfortunately, it has now been found by the present inventors that, when utilizing polyols other than ethylene oxide ("EO")-capped polyols. The reactivity of the polyols made with DMC catalysts is less than otherwise might be desired in the subsequent reaction of the polyol with an isocynaria to produce a polyure/thane.

A solution to this problem of less-than-desired reactivity would be highly desired by the DMC-catalyzed polyol manufacturing community. The present invention provides one solution to this problem.

In one aspect, the present invention relates to a process for enhancing the reactivity of a polyol free of ethylene oxide capping produced using a double metal cyanide catalyst which comprises incorporating into said polyol between 0.5 and 10 ppm based on the weight of said polyol of a base selected from an alkali metal hydroxide, alkalin eath metal alkoxide, alkaline earth metal alkoxide, or a combination thereof.

In another aspect, the present invention relates to a process for producing a base-containing polyol having enhanced reactivity toward polyisocyanates which comprises the steps of:

(a) Torming a polyol having a backbone comprising moieties selected from the group comprising athylene axide, propylene axide, and combinations thereot, said polyol being produced using a double metal cyanide catalyat and being free of ethylene axide capping and removing any residual double metal cyanide catalyst by treating the notivol with hydrogene nexide.

(b) incorporating into said polyol between 0.5 and 10 ppm based on the weight of said polyol of a base as described above.

These and other aspects will become apparent upon reading the following detailed description of the invention. It has now been surpisingly found that the reactivity of polyols made using a DMC catalyst toward polyols is significantly improved by incorporation of at least a trace amount of base into the polyol prior to the reaction of the polyol with a polyisocyanate to form a polyverthane. The resulting polyverthanes exhibit improved physicial properties, e.g., improved tensile strength, elongation and/or least returned, as compared to polyverthanes prepared using a DMC catalyst but not containing at least a trace amount of the base, as demonstrated by the working examples provided hereinbelow.

The DMC catalyst-prepared polyols of the present invention contain 0.5-10 ppm of a base, most preferably KOH or NaCH although other bases such as other alkali metal hydroxides, alkaline earth metal hydroxides, alkaline although such preferably at least 1.0 ppm, most preferably at least 1.5 ppm, of the base based upon the weight of the polyol are used. The amount of the base does not exceed 10 ppm (preferably not more than 5 ppm) based upon the weight of the polyol from the base-containing polyols prepared according to the present invention are particularly useful in the production of polyurethane sealants and elastomers, including thermoplastic and thermoset leastomers, as well as cast is leastomers, although they also are suitably utilized in the production of polyurethane subsor are suitably utilized in the production of polyurethane subsor are suitably utilized in the production of polyurethane subsor are suitably utilized in the production of polyurethane subsores, as well as a may be desired.

The polyurethanes made using the base-containing polyols prepared according to the present invention may be made by the prepolymer process or the one-shot process. The polyurethane isocyanate-terminated prepolymer that is utilized when employing the prepolymer process is prepared by reacting an organic polyisocyanate with a poly-alikylene either polyol(s) in an equivalent ratio of NOO to OH groups of from about 1.02/1 to about 15/1, using standard procedures, to yeld an isocyanate-terminated prepolymer of controlled molecular weight. Preferably, the NCO/OH ratio ranges from about 1.3/1 to about 5/1. The reaction may be accelerated by employing a catalyst; common urethane catalysts are well known in the art and include numerous organometallic compounds as well as amines, e.g., targines and metal compounds such as lead octoates, mercur's succinates, stannous octoate or dibutyltin diaurate may be used. Any catalytic amount may be employed; illustratively, such amount varies, depending on the particular catalyst utilized, from about 0.1 to about 1 precent by weight of the polyurethane prepolymer.

Preferred base-containing polyols within the scope of the present invention are the polyether diols and the polyether triols, and combinations thereof. Suitable polyether triols include various polyoxyalitylene polyols and mixtures thereof. These can be prepared, according to well-known methods, by condensing an alkylene oxide, or a mixture of alkylene oxides using random or step-wise addition, with a polyhydric initiator or mixture of initiators. Illustrative alkylene oxides

include thylen oxide, propylene oxide, butylene oxide, amylene oxide, aralkylene oxides such as styrene oxide, and the halogenated alkyl ne oxides such as trichlorobutylene oxide and so forth. The most preferred alkyl ne oxide is propylene oxide or a mixture thereof with thyl no oxide using random or step-wise oxyalkylation.

The polyhydric initiator us d in preparing the polyether triol reactant includes the following and mixtures thereof: the aliphatic triols such as glycerol, propoxylat d glycerol adducts, trimethylolpropan triethylolpropane, trimethylolpropane.

A preferred group of polyhydric initiators for use in preparing the polyether triol reactant is one which comprises triols such as glycerol, propoxylated glycerol adducts, trimethylolpropane and the like.

The polyether diols are prepared by an analogous reaction of alkylene oxide, or mixture of alkylene oxides with a polyhydric initiator. In this case the initiator is a diol such as ethylene glycol, 1,3-propylene glycol, dipropylene glycol, butane diols, pentane diols, and the like.

A preferred group of polyhydric initiators for use in preparing the polyether diol reactant is one which comprises diols such as ethylene glycol, diethylene glycols, propylene glycol, dipropylene glycol, or water.

The alkylene oxide-polyhydric initiator condensation reaction is carried out in the presence of a double metal cyanide catalyst. The use of a double metal cyanide catalyst rather than a base such as KCH during the preparation of the polylor provides distinct advantages in regard to the end group unsaturation level of the polyd produced. Without wishing to be bound by any particular theory, it is speculated by the present inventor that unsaturated end groups result in monofunctional species that act as chain stoppers in elastomer formation. In polyol synthesis with KCH catalysis the unsaturation formed increases as a direct function of equivalent weight. Eventually conditions are established wherein further propylene oxide addition fails to increase the molecular weight. In other words the use of alkali catalysts to produce high molecular weight, hydroxy terminated polyoxypropylene ethers results in a substantial toss in hydroxy functionality. With double metal cyanide catalysis much less unsaturation is formed allowing higher equivalent weight polyols to be prepared.

The double metal cyanide complex class catalysts suitable for use and their preparation are described in US-A-4,472,580, 4,477,589, 3,941,849, 4,242,490 and 4,335,188.

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One double metal cyanide complex catalyst found particularly suitable for use is a zinc hexacyanometallate of formula:

wherein M may be Co(III), or Cr(III) or Fe(II) or Fe(III); x, y, and z may be fractional numbers, integers, or zero and vary depending on the exact method of preparation of the complex.

In producing a polyurethane utilizing the base-containing polyols of the present invention, any suitable organic polyiscoryanate, or mixture of polyiscoryanate, are be used. Illustrative are toluene discoryanate, such as the 80:20 and the 85:35 mixtures of the 2,4- and 2,6-isomers, ethylene discoryanate, propylene discoryanate (Aphenyl) isocyanate (also referred to as diphenylmethane discoryanate or MDI), xylene discoryanate (XDI), isophorona discoryanate (IPDI), 33-bistoluene, 44-discoryanate, haxamethylene discoryanate (XDI), hydrogenated MDI, nydrogenated MDI, and modified liquid MDI adducts such as carbodimide-modified MDI, naphthalene-1,5-discoryanate, here polymeric isocyanates (XDI), and modified isophoronated MDI adducts such as carbodimide-modified MDI, naphthalene-1,5-discoryanate, and the like. In accordance with a particularly preferred embodiment of the invention, there is employed an isomeric mixture of 2,4- and 2,6-diouene discoryanate in which the weight ratio of the 2,4-isomer to the 2,6-isomer is from about 60:34 to about 90:20, as well as MDI.

Chain extenders which can be utilized in preparing polyurethanes, particularly' coatings, sealants, adhesives and elastioners, utilizing the base-containing polyots of the present invention include diols and diamines such as 4.4 methylene bis/c-hiorcaniting) ("MOCA"), buttane diol, hexane diol, propylene glycot bisphaneol. A or polyalitylene oxide polyols with molecular weights between 100 - 2,000, or the prepolymer can be moisture cured with water. The chain extenders can also be triols such as glycerine, trimethylol propane, or propoxylated adducts of glycerine or trimethylol propane or amines like diethyticiuene diamine, ethylene diamine, substituted aromatic diamines such as the product commercially available as UNILINK 4200, a product of UOP, Inc, triisopropyl amine, methylene bis/contochloro-aniline), N.-bis/c1-yhdroxypropyl-aniline which is commercially available as ISONOL 100, a product of Dow Chemical Corp., and the like, and combinations thereof. Preferred chain extenders include MOCA, butane diol, trimethylol propane, diethyllotuene diamine, N.-bis/c2-hydroxypropyl-aniline, and combinations thereof. The chain extension can be conducted either in situ during the prepolymer formation or in a separate reaction step.

In preparing the polyurethanes utilizing the base-containing polyols of the present invention, the polyol(s), polyisocylanat(e), chain extender(s), and other components are reacted, typically under conditions of an elevated temperature. Urethane forming calleyts can be used as well as antioxidants or other antidegradants. The urethane-forming components may be mixed with the usual compounding ingredients, e.g. plasticizers, adhesion promoters, fillers and

pigments like clay, silica, furmed silica, carbon black, talc, phthalocyanine blue or gr en, TiO₂, U-V absorbers, MgCO₃, CaCO₃, and the like. The compounding ingradients, such as filters, are suitably employed in the elastomer in an amount of betw en 0 and about 75 weight percent based upon the weight of the elastomer. The polymerization reaction may be carried out in a single reaction (one-shot process), or in one or more sequential steps (prepolymer process). In the one-shot process, all the isocyanate-reactiv components are reacted similariaeously with the polyisocyanati. In such process, it is normal practice to blend all components except the polyisocyanate into a "B-side" mixture, which is then process, it is normal practice to blend all components except the polyisocyanate into a "B-side" mixture, which is then reacted with the polyisocyanate to form the polyurentane andior polyures elastomer. However, the order of mixing is not critical as long as the components do not undesirably react before all components are present. The reaction mixture is usually then placed in a mold and cured at a suitable temperature. The appearatus used for blending and molding is not especially critical. Hand mixing, conventional machine mixing, and the so-called reaction injection molding (RIM) equipment are all suitable. In the prepolymer process, all or a portion of one or more of the isocyanate reactive materials is reacted with a stoichiometric excess of the polyisocyanate-teactive materials to prepare the polyureal hearther or the chain extendor, or a mixture of

As used herein, the term "molecular weight" is intended to designate number average molecular weight. The term "equivalent weight" designates the molecular weight divided by the number of hydroxyl groups per molecule of polyol.

EXAMPLE 1

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A. Preparation of a Polyol with a Double Metal Cyanide Catalyst

A propoxylated glycerine precursor 100 g (133.6 eq. wt., 0.748 eq.) was added to a 1 liter autoclave. Zinc hoxacyanocolbate (0.1 g) was added and the autoclave was flushed with nitrogen three times. The mixture was heated to 100°C. Propylene oxide (30 g) was added and it reacted as evidenced by a drop in pressure. PO was fed into the reactor at rate to maintain the pressure below 30 psi and 609.0 g of propylene oxide was added within two hours. At this point 548 g of the mixture was removed to allow space for more epoxide leaving 162, grams in the reactor. An additional 340 grams of propylene oxide were fed into the reactor over a period of 1.5 hours to produce a polyol with a molecular weight of 10.000, hydroxyl number 16.8. Using a similar procedure, polyols with hydroxyl numbers between 16 and 60 were prepared. Diols were prepared by starting with dipropylene glycol based precursors instead of glycerine based precursors.

B. Polyol Treatment with Hydrogen Peroxide to Remove the DMC Catalyst Using the Process Described in US-A-5,099,075

A polyol (500.0 g) prepared as described in Example 1A containing residual DMC catalyst was heated to 100°C and 30% hydrogen peroxide (3.0g. 0.2 xt.% peroxide) was added. The mixture was heated at 100°C for one hour and then Celite (10.0 g, 2 xt.%) was added and the mixture was vacuum stripped for one hour and filtered. A clear polyol containing 0 ppm cobalt and 0 ppm zinc was obtained.

EXAMPLE 2

Sealants Prepared with DMC Catalyzed Polyol and Adjusted Reactivity DMC Catalyzed Polyol

A. Prepolymer Preparation: TDI-80 22.86 g, 0.282 eq. and a 10,000 molecular weight triol prepared as described in example 1 (270.0 g, OH no. 17.3, 0.0833 eq.) were added to a flask under nitrogen and heated to 80°C for 5 hours with stirring. The percentage of free NCO was measured and found to be 2.40%.

B. Sealant Proparation: A prepolymer prepared as described in example 24 (108.0 g. 0.0606 eq.), PCLY-G® 20-56 polyol (58.7 g. 1020 eq. wt. 0.0576 eq., 105 index), and dried fibrous talc (40.9 g) were mixed on a high speed mixer for several minutes. The mixture was degassed and T-10 stannous octoate catalyst 0.724 g was added. The mixture was stirred under vacuum for two minutes and then poured between glass plates with a 3,2 mm (1/8*) spacer and cured at 70°C for 16 hours. The sealant was aged at 20°C and 50% relative humidity for one week before testino.

C. Polyol Reactivity Adjustment: Low levels of KOH (0.935, 1.87, 2.82 ppm) were added to a 10,000 mw triol that was prepared as described in example one. The polyol was vacuum dried at 110°C for 2 hours and then used to prepare sealants as described above in example 2a & b. Properties obtained with the adjusted reactivity polyols



and th unadjusted polyol are compar d in Table 1 below:

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Shore A Hardness	'n	50	70	04	
Ult. Elongation		2390	3950	930	
Ult. Kensile		310	300	250	
Modulus 300% Elong.	To soft to test	11	20	164	
KOH added T-10 catalyst Modulus to polyol grams 300% Elon	0.724	0.743	0.73	0.66	
KOH added to polyol	0	0.935 ppm	1.87 ppm	2.82 ppm	

The above data show that when no base was added to the polyol a soft sealant (Shore A hardness 5) that was too soft to test was obtained. When small levels of base were added to the polyol sealants with higher hardness (20-40 Shore A) and excellent tensile elongation properties were obtained.

EXAMPLE 3

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Cast Elastomers Pr par d with DMC Catalyz d Polyols and with Adjusted Reactivity DMC Catalyzed Polyols

3A. A 2000 molecular weight diol was prepared with DMC catalyst using a propoxylated dipropylene glycol precurrence using the procedure described in Example la. The polyol was treated with hydrogen peroxide as described in example to and the cobalt and zinc were completely removed from the polyol.

The reactivity of the polyol was adjusted by adding varying levels of KOH (1.87, 2.81, 3.74 ppm). The polyols were vacuum dried at 110°C for two hours to remove water.

3B. Prepolymer preparation: Diphenyl methane diisocyanate (MDI) was melted in an over at 50°C. The polyol 400 g, 0.4 eq. were added to a flask under nitrogen and heated to 60°C. MDI (148.0 g, 1.18 eq.) was added and the mixture was heated to 80°C for 2.5 hours with stirring under nitrogen. The percentage of free NCO was measured and found to be 6%.

3C. Elastomer Preparation: The prepolymer described in example 3B above 200 g, 0.286 eq. and dried butane diol 12.5 g, 0.277 eq., 103 index and Cocure 30 catalyst 0.32 g were mixed on a high speed mixer for 15-20 seconds at 3000-4000 rpm. The mixture was degassed in a vacuum desicator until the surface was bubble free. The mixture was poured between glass plates using a 3.2 mm (1.87) spacer and cured in an oven at 100-105°C for 16 hours. The elastomers were aged at 50% relative humidity for one week before testing.

Physical properties of the elastomers obtained with the adjusted reactivity polyols compared to the unadjusted polyol are presented in Table 2 below.

TABLE II

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Physical Properties of Elastomers Prepared with Adjusted Reactivity DMC Catalyzed Polyols Compared to Unadjusted Polyol. MDI/2000 mw diol prepolymers, 6% free NCO, extended with butane diol.

Comp.	Set 1	33	38	32
Tear	Die-C	194	286	245
Elong.	**	479	850	654
Tensile	Strength	813	1684	2554
100%	Modulus	349	353	349
Shore A		02	89	89
ppm KOR added	to DMC polyol	0	1.87	2.81

The above data showed that small amounts of base added to the double metal cyanide catalyzed poly 1 dramatically improved the tensile strength, elongation and tear strength of the elastomers.

Claim

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- 1. A process for enhancing the reactivity of a polyol free of ethylene oxide capping produced using a double metal cyanide catalyst which is characterized by, after r moving any residual double metal cyanide catalyst by trating the polyol with hydrogen peroxide, incorporating into said polyol between 0.5 and 10 ppm, based upon the weight of said polyol, of a base selected from the group consisting of an alkali metal hydroxide, alkalin earth metal alkoxide, alkaline earth metal hydroxide, alkaline earth metal hydroxide, alkaline other host polyonized.
- 2. The process of claim 1 characterized in that said base is selected from the group consisting of an alkali metal hydroxide, alkaline earth metal alkoxide, alkaline earth metal alkoxide, and combinations thereof wherein said alkali metal is sodium or obtassium.
 - A process for producing a base-containing polyol having enhanced reactivity toward polyisocyanates which comprises the steps of:
 - (a) forming a polyol having a backbone comprising moieties selected from the group comprising ethylene oxide, propylene oxide, and combinations thereof, said polyol being produced using a double metal cyanide catalyst and being free of ethylene oxide capping, and removing any residual double metal cyanide catalyst by treating the polyol with hydrogen peroxide;
 - (b) incorporating into said polyol between 0.5 and 10 ppm, based on the weight of said polyol, of a base selected from the group consisting of an alkali metal hydroxide, alkali metal alkoxide, alkaline earth metal hydroxide, alkaline earth metal alkoxide, and combinations thereof.
 - 4. The process of claim 3 characterized in that said base is selected from the group consisting of an alkali metal hydroxide, alkalin metal alkoxide, alkaline earth metal hydroxide, alkaline earth metal alkoxide, and combinations thereof, wherein said alkali metal is soldium or obtained.

Patentansprüche

- Verfahren zur Erhöhung der Reaktivrität eines Polyols ohne Eltylenoxid-Kappe, das unter Verwendung eines Doppelmetallicyani-Katalysators hergestellt wurde, wobei das Verfahren dadurch gekennzeichnet ist, daß nach dem Entfemen von irgendwelchem restlichen Doppelmetallsyndi-Katalysator durch Behandeln das Polyols mit Wasserstoffperoxid in das Polyol zwischen 0,5 und 10 ppm, auf der Basis des Gewichts des Polyols, einer aus der aus einem Alkalimetallhydroxid. Alkalimetallalkoxid, Erdalkalimetallhydroxid, Erdalkalimetallalkoxid und Kombinationen davon bestehenden Gruppe aussewählten Base inkoppiert werden.
- Verlahren nach Anspruch 1, dadurch gekennzeichnet, daß die Base ausgewählt ist aus der aus einem Alkalimetallhydroxid, Alkalimetallalkoxid, Erdalkalimetallhydroxid, Erdalkalimetallalkoxid und Kombinationen davon bestehenden Gruppe, wobei das Alkalimetall Natrium oder Keilum ist.
 - Verfahren zur Herstellung eines Base enthaltenden Polyols mit erh\u00f6hter Reaktivit\u00e4t bez\u00fcglich Polyisocyanaten, das folgende Schritte aufweist:
- 45 (a) Herstellen eines Polyols mit einem Rückgrat, das aus der Ethylenoxid, Propylenoxid und Kombinationen davon aufwalsenden Gruppe ausgewählte Einheiten enthäti, whoel das Polyol unter Verwendung eines Doppelmetallcyanid-Katalysators hergestellt wird und frei von Ethylenoxid-Kappen ist, und Entfernen von irgendweichem restlichen Doppelmetallcyanid-Katalysator durch Behandeln des Polyols mit Wasserstoffperoxid, (b) Inkorporieren von zwischen O. 5 und 10 ppm, auf der Basis des Gewichts des Polyols, einer sa der aus einem Alkalimetallhydroxid, Alkalimetallalkoxid, Erdalkalimetallhydroxid, Alkalimetallalkoxid, Erdalkalimetallhydroxid, Pradikalimetallalkoxid und Kombinationen davon bestehenden Gruppe ausgewählten Base in das Polyol.
 - 4. Vorlahren nach Anspruch 3. dadurch gekennzeichnet, daß die Base ausgewählt ist aus der aus einem Alkalimetalihydroxid, Alkalimetallakoxid, Erdalkalimetallihydroxid, Erdalkalimetallakoxid und Kombinationen davon bestehenden Gruppe, wobei das Alkalimetall Natrium oder Kalium ist.

R vendications

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- 1. Procédé pour augmenter la réactivité d'un polyol ex mpt de coliflage par l'oxyde d'éthylène, produit par utilisati n d'un catalyseur à base d'un cyanure métalique doubl , qui st caractérisé en ce que, après avoir eliminé tout catalyseur la base d'un cyanure métalique double par traitement du polyol avec du peroxyde d'hydrogèn , il consiste à incorporer dans ledit polyol entre 0,5 et 10 ppm, par rapport au poids dudit polyol, d'une base choisie dans le groupe comprenant les hydroxydes de métaux alcalins, les alkylates de métaux alcalins, les hydroxydes de métaux alcalins, les put scandins valicalins, les hydroxydes de métaux alcalins les alkylates de métaux alcalins, les hydroxydes de métaux alcalins les metaux alcalins, les hydroxydes de métaux alcalins les metaux alcalins les metaux alcalins les metaux alcalins.
- 9 2. Procédé selon la revendication 1, caractérisé en ce que ladite base est choisie dans le groupe comprenant les hydroxydes de métaux alcalins, les alkylates de métaux alcalins, les hydroxydes de métaux alcalino-dereux, les alkylates de métaux alcalino-terieux et leurs combinations, où ledit nétal alcalin et le sodium ou le potassium.
- Procédé pour produire un polyol contenant une base, présentant une réactivité augmentée vis-à-vis des polyisocyanates, qui comprend les étapes consistant :
 - (a) à tormer un polyoi ayant un squelette comprenant dos fragments choisis dans le groupe comprenant l'oxyde d'éthylène, l'oxyde de propylène et leurs combinaisons, ledit polyoi étant produit par utilisation d'un catalyseur à base d'un cyanure métallique double, et étant exempt de coffiage par de l'oxyde d'éthylène, et à éliminer tout catalyseur résiduel à base d'un cyanure métallique double, par traitement du polyoi avec du peroxyde drivdrocène :
 - (b) à incorporer dans ledit polyol de 0,5 à 10 ppm, par rapport au poids dudit polyol, d'une base choisie dans le groupe comprenant les hydroxydes de métaux alcalins, les alkylates de métaux alcalins, les hydroxydes de métaux alcalino-terreux, les alkylates de métaux alcalino-terreux et leurs combinaisons.
- 4. Procédé selon la revendication 3, caractérisé en ce que ladite base est choisie dans le groupe comprenant les hydroxydes de métaux aicalims, les alkylates de métaux aicalins, les hydroxydes de métaux a baino-terreux, les alkylates de métaux aicalimo-terreux et leurs combinaisons, oi ledit métal abain set le sodium ou le potassium.